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ELECTRICALLY INSULATING, HIGH PRESSURE SEALS FOR INTERNAL HEATED PRESSURE VESSELS

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INTRODUCTION

The physical properties of all materials change as either their temperature or pressure or both are changed. As a consequence, both homogeneous and heterogeneous chemical equilibrium are a function of temperature and pressure. The control and manipulation of these variables is essential to many scientific studies. Geologists have a special interest in the simultaneous application of temperature and pressure as a means of duplicating the conditions in planetary interiors.

One of the types of apparatus used to perform such studies is the internally heated pressure vessel (IHPV), in which temperatures as high as 1500°C can be attained at hydrostatic pressures up to 10 kilobars. The basic concept of the IHPV was developed by P. W. Bridgman (ref. 1) and the various applications of IHPV systems to geological problems are presented by Holloway, who also describes most of the techniques in current use (ref. 2). Holloway's article also provides access to most of the early work on IHPV systems.

In brief, IHPV systems employ a large diameter, cylindrical, steel pressure vessel with the furnace inside the vessel. It is this basic concept--place the source of heat inside the pressure container so that the vessel is not weakened by heating--which permits the system to operate at such high temperatures and pressures simultaneously. It is also the source of one of the most frustrating problems of IHPV systems: the provision of electrically insulated feed-throughs to supply power to the furnace while the integrity of the pressure system is maintained. This report describes an electrode seal which overcomes most of the problems of previous designs.

ELECTRODE SEALS

In most designs, the insulators for the furnace electrode were machined from limestone and crushed into place using an arbor press. Although a good seal can be made this way, the machining of the limestone is difficult. Also, during depressurization the release of gas occluded in the insulator at high pressure almost always rips the insulator apart, and thus new insulators must be made for every experiment.

In order to overcome these problems, we have adapted Bridgman's design for the main pressure seals to the electrode seal. The main pressure seal (ref. 1) uses thin washers of rubber, lead, brass, and steel which are extruded by pressure and form a pressure seal by pressing against the walls of the vessel. We have used washers made of insulators--neoprene, teflon, nylon, and alumina (better than 98% Al_2O_3) ceramic--to make our seals. Our design is shown in figure 1. These materials work well together because the neoprene, teflon, and nylon extrude at successively higher pressures providing just the right amount of sealing while the alumina ceramic provides the mechanical strength necessary to support the seal and position the electrode assembly.

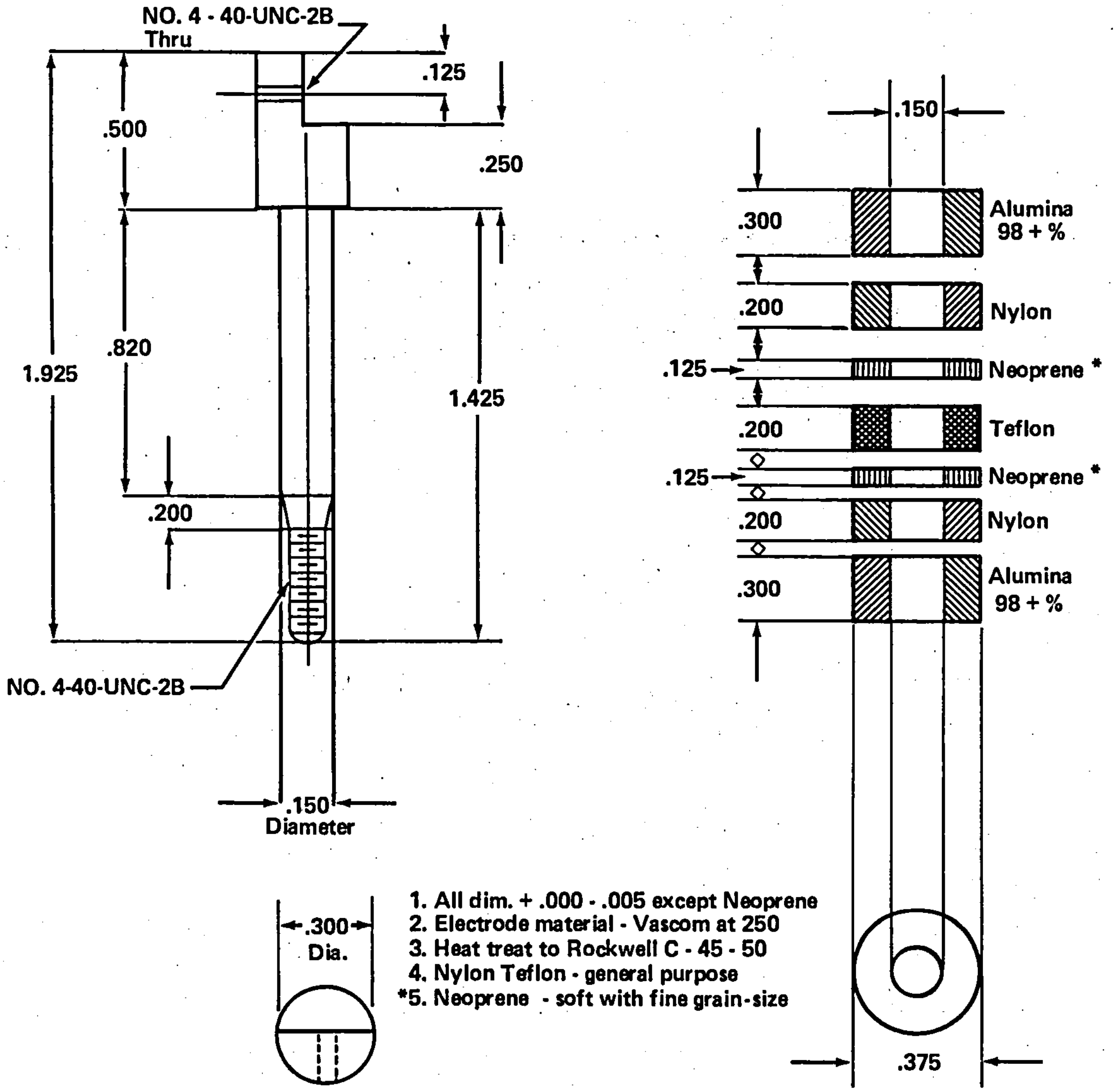

Figure 1. IHPV furnace electrode and pressure seal. Neither the pressure vessel head nor the brass extensions are shown. Base extension attaches to the nipple at the bottom of the electrode; the rod and the cone portion of the electrode are insulated with shrink sleeving. Dimensions are shown as + 0.000, - 0.005 except neoprene which is simply cut to fit.

All of the parts are machined or cut to the sizes and tolerances shown in figure 1. The parts should fit together snugly. The electrode must be made from hardened steel (RC40-50). If a softer material is used, the electrode will pinch off as the seal assembly extrudes and be ejected like a missile. The washers are placed into the feed-through hole in the pressure head in the sequence shown in figure 1. An extension rod (brass in our system) is attached to the nipple on the end of the electrode and is insulated with shrink sleeving. The electrode is slipped into the washers using an arbor press to apply the minimum force necessary to insert it. If too much force is used, the alumina ceramic will fracture; this must be avoided if the seals are to work properly.

The pressure head is then fitted into the pressure vessel and the system pressurized. Before use, it is necessary to pressurize the system to at least 2 kilobars in order to properly seat the electrode and extrude the seals. After that, use is routine.

EXPERIENCE AND CAUTIONS

We have used the seals to pressures of 116,000 psi (8 kilobars) at furnace temperatures up to 1250°C. The maximum pressure at which they can be used has not been determined, and thus use beyond the conditions we site should be approached with extreme caution.

We have been able to use the seals for several months up to twenty pressurization-depressurization cycles before replacement was necessary. However, we have no data from which a general life cycle history for the seals can be constructed; again, some caution and discretion is advised.

The grade of neoprene is critically important. Fine-grained, high quality neoprene is required. All neoprenes contain some elemental carbon; under high pressure these carbon grains are forced together and can form an electrical short. We have found this to be a problem only with coarse grained varieties of neoprene.

Finally, the teflon and nylon seals will soften and fail if they are heated. Consequently, it is essential that the vessel be well cooled and that the ends of the furnace be packed with an insulator (e.g., silica wool) to prevent radiative and conductive transfer of heat to the pressure heads.

CONCLUSION

Neoprene, nylon, teflon, and alumina washers have been used to construct an electrically insulating, high pressure seal for internally heated pressure vessel systems. These seals have considerable advantages over conventional designs because they are easier to make and they endure multiple experiments. This design is based on Bridgman's design for the main pressure seals. Thus,

they appear to be the solution to one of the most frustrating and time-consuming aspects of work with IHPV systems.

These seals have been used routinely with a minimum of problems for several years at pressures up to 116,000 psi (8 kilobars) at process temperatures as high as 1250°C.

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1. Bridgman, P. W. (1949). The Physics of High Pressures. London: G. Bell and Sons.

2. Holloway, J. R. (1971). "Internally Heated Pressure Vessels," in Research Techniques for High Pressure and High Temperatures. (Gene C. Ulmer, ed.) New York: Springer-Verlag, pp. 218-258.

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